Nitrogen, Ammonia

★Method 8038

Nessler Method[1]

(0.02 to 2.50 mg/L NH_3–N)

Scope and Application: For water, wastewater, and seawater; distillation is required for wastewater and seawater; USEPA accepted for wastewater analysis (distillation required); see Distillation on page 4 of this procedure.

[1] Adapted from *Standard Methods for the Examination of Water and Wastewater 4500-NH_3 B & C.*

Test Preparation

Before starting the test:

For more accurate results, determine a reagent blank value for each new lot of reagent. Follow the procedure using deionized water instead of the sample. Subtract the reagent blank value from the final results or perform a reagent blank adjust. See the user manual for more information.
Nessler Reagent contains mercuric iodide. Both the sample and the blank will contain mercury (D009) at a concentration regulated as a hazardous waste by the Federal RCRA. Do not pour these solutions down the drain. Refer to a current MSDS for safe disposal and handling instructions.
If using the Pour-Thru Cell, periodically clean the cells by pouring a few sodium thiosulfate pentahydrate crystals into the cell funnel. Flush with enough deionized water to dissolve. Rinse the cell thoroughly.

Collect the following items:	**Quantity**
Ammonia Nitrogen Reagent set	1
Deionized Water	25 mL
Graduated Mixing Cylinders	2
Sample Cells, 1-inch square, 10-mL	2
Serological Pipet, 1-mL	2

Note: *Reorder information for consumables and replacement items is on page 5.*

Note: *Nessler Reagent is toxic and corrosive. Pipet carefully, using a pipet filler. When dispensing reagent from a dropper bottle, hold the bottle vertically. Do not hold the bottle at an angle.*

Note: *A yellow color will develop if ammonia is present. (The reagent will cause a faint yellow color in the blank.)*

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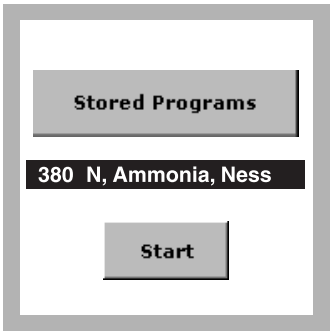
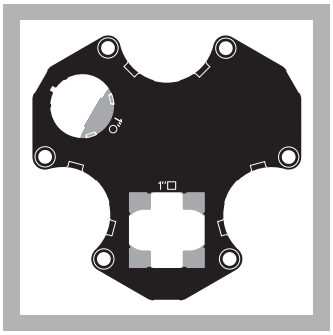
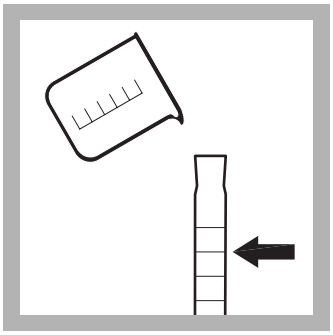


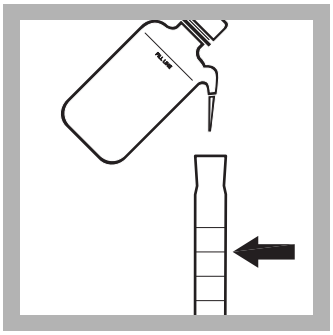
1. Select the test.



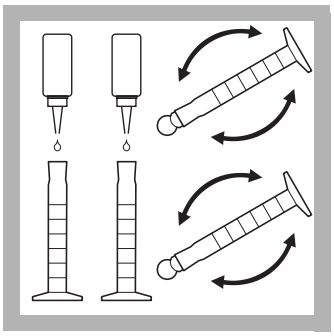
2. Insert the Multi-cell Adapter with the 1-inch square cell holder facing the user.



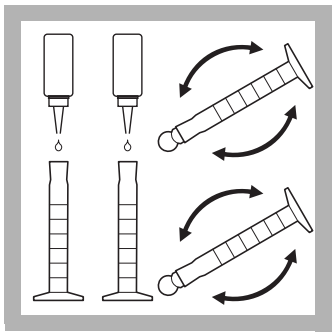
3. Prepared Sample: Fill a 25-mL mixing graduated cylinder to the 25-mL mark with sample.



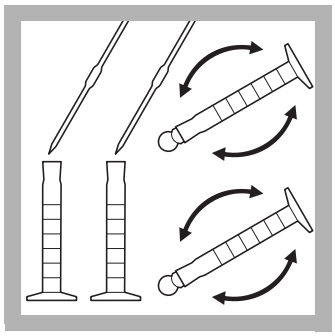
4. Blank Preparation: Fill a 25-mL mixing graduated cylinder to the 25-mL mark with deionized water.



5. Add three drops of Mineral Stabilizer to each cylinder. Stopper and invert several times to mix.



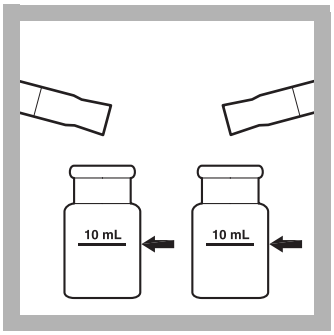
6. Add three drops of Polyvinyl Alcohol Dispersing Agent to each cylinder. Stopper and invert several times to mix.



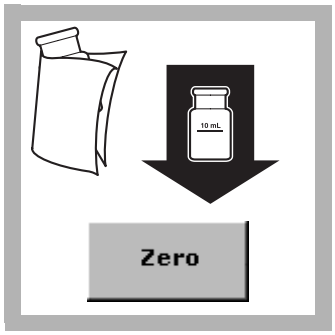
7. Pipet 1.0 mL of Nessler Reagent into each cylinder. Stopper and invert several times to mix.

8. Press **TIMER>OK**.

A one-minute reaction period will begin.



9. Pour 10 mL of each solution into a square sample cell.



10. When the timer expires, insert the blank into the cell holder with the fill line facing the user. Press **ZERO**. The display will show:
0.00 mg/L NH_3 –N

11. Wipe the prepared sample and insert it into the cell holder with the fill line facing the user.

Results are in mg/L NH_3–N.

Interferences

Table 1 Interfering Substances and Levels

Interfering Substance	Interference Levels and Treatments
Chlorine	Remove residual chlorine by adding 2 drops of sodium arsenite for each mg/L chlorine (Cl_2) from a 250 mL sample. Sodium thiosulfate can be used instead of sodium arsenite. See Sample Collection, Storage, and Preservation.
Hardness	A solution containing a mixture of 500 mg/L $CaCO_3$ and 500 mg/L Mg as $CaCO_3$ does not interfere. If the hardness concentration exceeds these concentrations, add extra Mineral Stabilizer.
Iron	Interferes at all levels by causing turbidity with Nessler Reagent.
Seawater	May be analyzed by adding of 1.0 mL (27 drops) of Mineral Stabilizer to the sample before analysis. This complexes the high magnesium concentrations found in sea water, but the sensitivity of the test is reduced by 30 percent due to the high chloride concentration. For best results, perform a calibration, using standards spiked to the equivalent chloride concentration, or distill the sample as described below.
Sulfide	Interferes at all levels by causing turbidity with Nessler Reagent.

Table 1 Interfering Substances and Levels (continued)

Interfering Substance	Interference Levels and Treatments
Glycine, various aliphatic and aromatic amines, organic chloramines, acetone, aldehydes and alcohols	May cause greenish or other off colors or turbidity. Distill the sample if these compounds are present.

Sample Collection, Storage, and Preservation

Collect samples in clean glass or plastic bottles. If chlorine is present, add one drop of 0.1 N Sodium Thiosulfate* for each 0.3 mg/L Cl_2 in a 1-liter sample. Preserve the sample by reducing the pH to 2 or less with sulfuric acid (at least 2 mL). Store at 4 °C (39 °F) or less. Preserved samples may be stored up to 28 days. Warm samples to room temperature and neutralize with 5 N Sodium Hydroxide* before analysis. Correct the test result for volume additions.

Accuracy Check

Standard Additions Method (Sample Spike)

1. After reading test results, leave the sample cell (unspiked sample) in the instrument.

2. Press **OPTIONS>MORE**. Press **STANDARD ADDITIONS**. A summary of the standard additions procedure will appear.

3. Press **OK** to accept the default values for standard concentration, sample volume, and spike volumes. Press **EDIT** to change these values. After values are accepted, the unspiked sample reading will appear in the top row. See the user manual for more information.

4. Snap the neck off a Nitrogen Ammonia Voluette® Ampule Standard, 50-mg/L NH_3–N.

5. Prepare three sample spikes. Fill three mixing cylinders with 25 mL of sample. Use the TenSette® Pipet to add 0.1 mL, 0.2 mL, and 0.3 mL of the 50 mg/L standard, respectively, to each sample and mix thoroughly.

6. Analyze each sample spike as described in the procedure above, starting with the 0.1 mL sample spike. Accept each standard additions reading by pressing **READ**. Each addition should reflect approximately 100% recovery.

7. After completing the sequence, press **GRAPH** to view the best-fit line through the standard additions data points, accounting for the matrix interferences. Press **IDEAL LINE** to view the relationship between the sample spikes and the "Ideal Line" of 100% recovery.

Standard Solutions Method

1. To check accuracy, use a 1.0-mg/L Nitrogen Ammonia Standard Solution. Or, prepare a 1.0-mg/L ammonia nitrogen standard solution by pipetting 1.00 mL of Nitrogen Ammonia Voluette® Ampule Standard, 50-mg/L, into a 50-mL volumetric flask. Dilute to the mark with deionized water. Prepare this solution daily. Perform the Nessler procedure as described above.

2. To adjust the calibration curve using the reading obtained with the standard solution, press **OPTIONS>MORE** on the current program menu. Press **STANDARD ADJUST**.

* See Optional Reagents and Apparatus on page 5.

3. Press **ON**. Press **ADJUST** to accept the displayed concentration. If an alternate concentration is used, press the number in the box to enter the actual concentration, then press **OK**. Press **ADJUST**.

Method Performance

Precision
Standard: 1.00 mg/L NH_3–N

Program	95% Confidence Limits of Distribution
380	0.99–1.01 mg/L NH_3–N

Sensitivity

Portion of Curve	ΔAbs	ΔConcentration
Entire range	0.010	0.02 mg/L NH_3–N2

Distillation

1. Measure 250 mL of sample into a 250-mL graduated cylinder and pour into a 400-mL beaker. Destroy chlorine, if necessary, by adding 2 drops of Sodium Arsenite Solution per mg/L Cl_2.

2. Add 25 mL of Borate Buffer Solution and mix. Adjust the pH to about 9.5 with 1 N sodium hydroxide solution. Use a pH meter.

3. Set up the General Purpose Distillation Apparatus as shown in the *Distillation Apparatus Manual*. Pour the solution into the distillation flask. Add a stir bar.

4. Use a graduated cylinder to measure 25 mL of deionized water into a 250-mL Erlenmeyer flask. Add the contents of one Boric Acid Powder Pillow. Mix thoroughly. Set the flask under the still drip tube. Elevate so the end of the tube is immersed in the solution.

5. Turn on the heater power switch. Set the stir control to 5 and the heat control to 10. Turn on the water and adjust to maintain a constant flow through the condenser.

6. Turn off the heater after collecting 150 mL of distillate. Immediately remove the collection flask to avoid sucking solution into the still. Measure the distillate to ensure 150 mL was collected (total volume = 175 mL).

7. Adjust the pH of the distillate to about 7 with 1 N sodium hydroxide. Use a pH meter.

8. Pour the distillate into a 250-mL volumetric flask; rinse the Erlenmeyer with deionized water. Add the rinsings to the volumetric flask. Dilute to the mark. Stopper. Mix thoroughly. Analyze as described above.

Summary of Method

The Mineral Stabilizer complexes hardness in the sample. The Polyvinyl Alcohol Dispersing Agent aids the color formation in the reaction of Nessler Reagent with ammonium ions. A yellow color is formed proportional to the ammonia concentration. Test results are measured at 425 nm.

Consumables and Replacement Items

Required Reagents

Description	Quantity/Test	Unit	Cat. No.
Ammonia Nitrogen Reagent Set, includes:	—	—	24582-00
Nessler Reagent	2 mL	500 mL	21194-49
Mineral Stabilizer	6 drops	50 mL SCDB	23766-26
Polyvinyl Alcohol Dispersing Agent	6 drops	50 mL SCDB	23765-26
Water, deionized	25 mL	4 L	272-56

Required Apparatus

Description	Quantity/Test	Unit	Cat. No.
Cylinder, graduated, mixing, 25-mL	2	each	20886-40
Pipet, serological, 1-mL	2	each	9190-02
Pipet Filler, safety bulb	1	each	14651-00
Sample Cells, 1-inch square, 10-mL, matched pair	2	2/pkg	24954-02

Recommended Standards and Apparatus

Description	Unit	Cat. No.
Flask, volumetric, Class A, 50 mL	each	14574-41
Nitrogen, Ammonia Standard Solution, 1-mg/L NH_3–N	500 mL	1891-49
Nitrogen, Ammonia Standard Solution, 10-mL Voluette® Ampule, 50-mg/L NH_3–N	16/pkg	14791-10
Pipet, TenSette® 0.1 - 1.0 mL	each	19700-01
Pipet Tips, for TenSette Pipet 19700-01	50/pkg	21856-96
Pipet Tips, for TenSette Pipet 19700-01	1000/pkg	21856-28
Pipet, volumetric, Class A, 1.00 mL	each	14515-35
Wastewater, Effluent Inorganics, for NH_3–N, NO_3–N, PO_4, COD, SO_4, TOC	500 mL	28332-49

Optional Reagents and Apparatus

Description	Cat. No.
Distillation Apparatus, General	22653-00
Heater and Support Apparatus, 115 VAC, 60 Hz	22744-00
Heater and Support Apparatus, 230 VAC, 50 Hz	22744-02
Mixing Cylinders	20886-40
Pour-Thru Cell Kit	LZV479
Sodium Thiosulfate, 0.1 N	323-32
Sodium Hydroxide, 5 N	2450-32

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